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ACCURACY OF THREE METHODS FOR DETERMINATION OF OXYGEN IN POTASSIUM AT CONCENTRATIONS LESS THAN 20 PARTS PER MILLION

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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SUMMARY

In order to assess the accuracy of the vacuum amalgamation, vacuum distillation, and neutron activation methods in the determination of oxygen in potassium at concentrations less than 20 parts per million, a number of comparative analyses were made on four different batches of potassium by a replicate sampling technique.

The environmental contamination pickup for the vacuum amalgamation and vacuum distillation methods, as a function of the pressure in the analytical system, was found to be 400 and 270 milligrams of oxygen per torr, respectively, independent of sample size. The method blanks were found to be 3.1 and 2.8 micrograms of oxygen, respectively.

Agreement between vacuum distillation and vacuum amalgamation mean oxygen values (corrected for systematic errors) in all cases was within 2 parts per million. The overall standard deviations of the vacuum distillation and vacuum amalgamation methods were 4.3 and 3.1 parts per million, respectively.

It was proposed that the difference found between neutron activation and the amalgamation (or distillation) method mean oxygen values was due in part to alkaline impurities other than potassium monoxide, for example, potassium carbonate, and in part to unaccounted-for contamination of the neutron activation samples. The effect of alkaline impurities on the analytical results of the neutron activation and amalgamation (or distillation) methods was calculated in terms of R, the ratio of potassium monoxide to other alkaline impurity, and f, a function of the difference in analytical values. Limiting-value estimates were made for both R and the environment contamination pickup in the neutron activation method.

When adjusted for the effect of alkaline impurities and systematic errors, oxygen values for all three methods showed good agreement.

INTRODUCTION

A need for the accurate determination of oxygen in potassium at low concentration has arisen out of the projected use of potassium as a fluid in space power systems (ref. 1), especially in view of the effect of oxygen impurities in promoting corrosion (ref. 2).

Currently, three principal methods are employed in the determination of oxygen in alkali metals: amalgamation, distillation, and neutron activation. Little is known, however, of the comparative accuracy and precision of these methods. The most widely applied method, amalgamation, has been used in the analyses of oxygen in sodium, potassium, and sodium-potassium alloy (NaK) (refs. 3 to 9). The distillation method has been employed primarily in the determination of oxygen in sodium (refs. 10 to 14). Neither method is specific for oxygen; the vacuum amalgamation and vacuum distillation methods culminate in the isolation, respectively, of a mercury insoluble or of a nonvolatile alkaline residue followed by alkalimetry of the residue. A recently introduced method, neutron activation, has been used for the determination of oxygen in potassium (ref. 15) and cesium (refs. 16 and 17). This method, although specific for oxygen, cannot distinguish among the several oxygen species that may be present, such as alkali metal carbonate, monoxide, or superoxide.

The purpose of this investigation was to compare the analytical results of the vacuum amalgamation, vacuum distillation, and neutron activation methods on replicate samples of potassium containing, nominally, less than 20 parts per million oxygen. Agreement of results could be taken as an index of accuracy; nonagreement could be used to establish a quantitative relation among methods.

Before results can be compared, however, the systematic errors inherent to each method had to be ascertained and the raw data corrected accordingly. As described herein, experimental values were obtained for the errors associated with the vacuum distillation and vacuum amalgamation methods, and limiting value estimates were made of the errors associated with the neutron activation method. For the purpose of experimental determination, the systematic error for each method was separated into two parts: (1) the error resulting from contamination of the sample by impurity gases present in the analytical environment and (2) all remaining errors, referred to herein as the method blank. The environmental contamination error was obtained from the slope of a plot of oxygen-found values against system pressure. The method blank was obtained from the intercept of a plot of environmental-contamination-corrected oxygen found against sample weight.
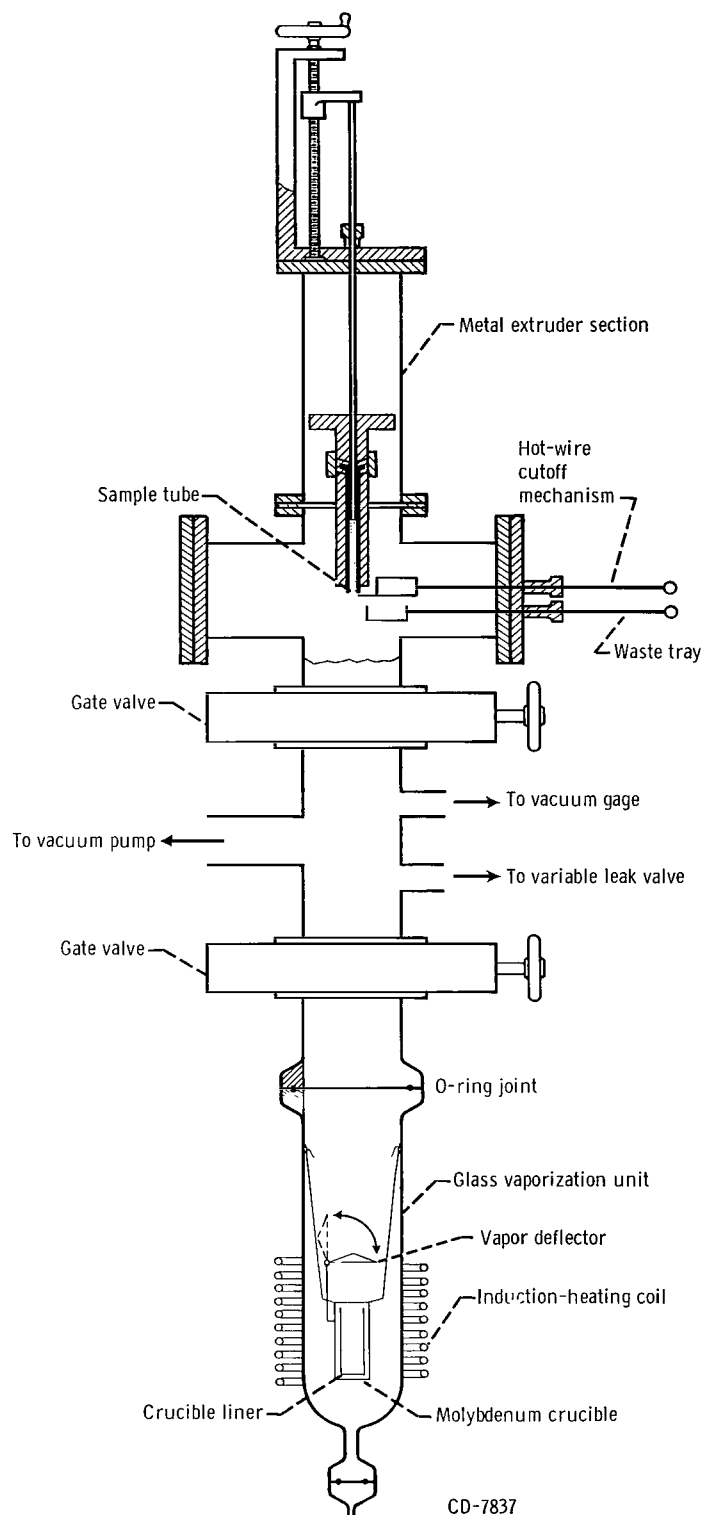
Metal extruder section

Hot-wire
cutoff
mechanism

Sample tube

Waste tray

Gate valve

To vacuum gage

To vacuum pump

To variable leak valve

Gate valve

O-ring joint

Glass vaporization unit

Vapor deflector

Induction-heating coil

Crucible liner

Molybdenum crucible

CD-7837

Figure 1. - Analytical apparatus for vacuum distillation of alkali metals.

EXPERIMENTAL APPARATUS AND PROCEDURE

Potassium Samples

The potassium used for this study was filtered, hot-gettered, and finally vacuum distilled, as described in reference 18. Four separate 1-pound batches of potassium were prepared in this manner and are herein designated as series I, II, III, and IV. Following the purification operations, a number of $\frac{3}{8}$-inch-outside-diameter by $6\frac{3}{4}$-inch stainless steel sample tubes were filled simultaneously with potassium of a given series, capped, and stored in vacuum until used (ref. 18). In all the analyses, samples were taken by extrusion of the potassium from the sample tubes (ref. 4).

Vacuum Distillation

Apparatus. - The vacuum distillation apparatus shown in figure 1 consists of an extruder section, a pressure monitoring section, and a vaporization section. The middle section is connected to a 2-inch vacuum diffusion pump through 2-inch-diameter tubing. A liquid-nitrogen-cooled baffle is used above the diffusion pump. The vaporization unit, made of heat-resistant glass, is 12 inches in length and $2\frac{1}{2}$ inches in diameter, and is connected to the metal system with an O-ring joint. A 1-inch-diameter, $2\frac{1}{2}$-inch-long molybdenum crucible containing a loose-fitting crucible liner of either nickel or glass is suspended with wires from glass hooks on the inside of the glass envelope. Above the crucible is suspended a hinged vapor deflector that can be swung up or down remotely with a magnet. A $2\frac{1}{2}$-kilowatt induction heater is used to heat the crucible, and the temperature of the crucible is measured by means of a Chromel-Alumel thermocouple inserted in a hole in the base of the crucible liner.

Procedure. - A sample tube filled with potassium was placed in the extruder, and the extruder section was evacuated. The crucible assembly was suspended inside the glass envelope, and the envelope was connected to the vacuum system and evacuated. The crucible was then outgassed at $400°$ to $500°$ C for at least 1 hour. After the crucible had cooled to room temperature and an operable vacuum of 7×10^{-6} torr or less was obtained, the analysis was begun.

A potassium sample was extruded, cut, and dropped into the distillation crucible. The vapor deflector was swung down and heat was applied to the crucible while the glass envelope was externally cooled by means of an air blower. In the temperature range $140°$ to $160°$ C vaporization of the potassium could be observed; at $200°$ C the vaporization rate was appreciable and the majority of the potassium sample had distilled. After several hours at $200°$ C, the crucible temperature was increased to $300°$ C for an additional

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2 or 3 hours to ensure complete potassium vaporization. Condensation of the potassium vapor took place on the inner surface of the glass envelope. The crucible vapor deflector acted as a baffle directing the potassium vapor to the lower half of the glass chamber.

At the completion of the distillation the crucible was allowed to cool, the glass chamber was backfilled with argon, and the crucible assembly was removed. The crucible liner was removed and rinsed four times with boiled distilled water, and the basic solution was titrated with 0.005 normal sulfuric acid (H_2SO_4) to the methyl red end point. The equivalent amount of oxygen was calculated with the assumption that the distillation residue was potassium monoxide (K_2O).

Initially the potassium sample weight was determined by slowly oxidizing the potassium on the walls of the glass chamber with water vapor and then titrating the basic solution with 0.1 normal H_2SO_4 to the methyl red end point. Subsequently, sample weight was calculated by measuring displacement of the extruder piston; a correlation of sample weight and displacement of the extruder piston, as measured by the number of turns of the extruder lead screw, yielded a value of 51.4±1.2 milligrams of potassium per extruder screw revolution.

Vacuum Amalgamation

The apparatus and procedure for the vacuum amalgamation method have been described in reference 4.

Neutron Activation

Neutron activation analyses on the potassium samples were performed by the General Dynamics Corporation (ref. 15). The copper capsule container employed in the series III analysis was filled at the Lewis Research Center with potassium from a sample tube and sealed by electron-beam welding in a vacuum of approximately 10^{-5} torr. The stainless-steel capsule used in the series IV analysis was filled with potassium from a sample tube and sealed at the General Dynamics Corporation in an inert gas chamber.

Environmental Contamination Study

Contamination of an analytical sample was assumed to be due mainly to real air leaks into the apparatus vacuum environment. To determine the effect of air contamination on the analytical results, vacuum distillation and vacuum amalgamation method

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analyses were performed at higher pressures than normal (up to 10^{-4} torr) by admitting dry air into the analytical system through a variable leak valve. The partial pressure of oxygen in the vacuum environment, measured with a mass spectrometer, varied directly with the total system pressure. The measured partial pressure of water vapor remained constant (approximately 3×10^{-6} torr) over the range of pressures investigated. Throughout each analysis, system pressure was maintained constant. Sample size, analysis time, and procedure were reproduced as closely as possible for analyses made using each method.

Calculations

The results for the vacuum distillation and vacuum amalgamation methods were calculated from the neutralization titration in the following manner:

$$\text{mg O} = \frac{(\text{ml acid for oxide})(0.005 \text{ N } H_2SO_4)(8 \text{ mg O/meq})}{(\text{ml acid for sample})(0.10 \text{ N } H_2SO_4)(39 \text{ mg K/meq})} \text{ mg K}$$

RESULTS

Data from the determination of oxygen in potassium by the vacuum amalgamation, vacuum distillation, and neutron activation methods are presented in table I. It is apparent from the values shown for the environmental contamination study runs (D I-1 and A I-6) that there is a marked effect of system pressure on the analytical results. The environmental contamination for the distillation and amalgamation methods was estimated by plotting the oxygen values found against the system pressure (fig. 2). The contamination pickups for the distillation and amalgamation methods were found to be 270 and 400 milligrams of oxygen per torr, respectively.

To ascertain the effect, if any, of sample size on contamination pickup, the series I data were treated as follows:

(1) Appropriate environmental contamination corrections were applied to the oxygen values by using the contamination pickup factors.

(2) The oxygen values corrected for environmental contamination were normalized, arbitrarily, to either a 0.514-gram or a 0.642-gram sample weight in the case of the distillation or amalgamation methods, respectively.

(3) The normalized oxygen values were plotted against the actual sample weight, and a regression analysis was performed to obtain the slope and standard deviation of the regression line.

TABLE I. - DETERMINATION OF OXYGEN IN POTASSIUM BY VACUUM DISTILLATION, VACUUM AMALGAMATION, AND NEUTRON ACTIVATION

Sample[a]	System pressure, torr	Potassium sample weight, g	Oxygen found uncorrected, μg	Oxygen found corrected for systematic errors[g] μg	ppm
Series I potassium					
[b]D I-1	7.0×10^{-6}	0.514	4.8	0.1	0.2
[b]D I-1	2.0×10^{-5}	.514	8.4	.2	.4
[b]D I-1	4.0	.514	13.6	0	0
[c]D I-2	4.0×10^{-6}	.765	12.8	8.9	11.6
[c]D I-2	2.6	.765	6.8	3.3	4.3
[c]D I-3	3.4	.756	10.0	6.3	8.3
[c]D I-3	3.5	1.512	15.9	12.2	8.1
D I-4	4.0	1.750	12.0	8.1	4.6
D I-4	3.5	1.800	10.4	6.7	3.7
D I-4	3.3	.257	4.4	.7	2.7
[d]D I-5	4.0	1.540	17.3	13.4	8.7
[d]D I-5	4.5	.360	9.2	5.2	14.4
[d]D I-5	5.5	.257	4.8	.5	1.9
[d]D I-5	5.5	.514	10.0	5.7	11.1
[b]A I-6	2.5×10^{-6}	.642	7.2	3.1	4.8
[b]A I-6	3.0×10^{-5}	.642	21.6	6.5	10.1
[b]A I-6	5.0	.642	25.2	2.1	3.3
[b]A I-6	1.0×10^{-4}	.642	50.0	6.9	10.7
[c]A I-3	1.0×10^{-5}	.840	12.4	5.2	6.3
[c]A I-3	8.5×10^{-6}	.945	14.8	8.3	8.8
[c]A I-2	8.0	1.280	15.2	8.9	7.0
Series II potassium					
[c]D II-1	5.0×10^{-6}	1.220	14.0	9.8	8.0
D II-2	6.0	.985	16.0	11.6	11.8
D II-2	5.0	.783	13.0	8.8	11.2
D II-2	5.0	.365	8.4	4.2	11.5
D II-2	7.0	1.298	14.0	9.3	7.2
D II-2	4.0	.518	7.2	3.3	6.4
D II-2	1.4	1.562	11.0	7.8	5.0
[c,e]A II-1	1.0×10^{-5}	1.00	12.0	4.9	4.9
[c,e]A II-1	2.0	1.41	19.7	8.6	6.1
[e]A II-3	9.0×10^{-6}	1.23	18.5	11.8	9.6
Series III potassium					
D III-1	4.0×10^{-6}	1.038	19.2	15.3	14.7
D III-1	3.0	1.022	13.2	9.6	9.4
[d]D III-1	4.0	.773	17.6	13.7	17.7
[d]D III-1	6.0	.780	22.0	17.6	22.6
[d]D III-1	4.0	.520	12.4	8.5	16.3
[e]A III-2	1.0×10^{-5}	1.69	42.3	35.2	20.8
[e]A III-2	4.0	1.72	44.7	25.6	14.9
[e]A III-3	2.0	.79	19.6	8.5	10.8
[e]A III-4	1.5	.90	23.5	14.4	16.0
[f]NA III-5		1.065	[h]60±1	[i]25±9	[i]23.5±8.4
Series IV potassium					
[e]A IV-1	6.0×10^{-6}	1.44	16.8	11.3	7.8
[e]A IV-1	8.0	1.54	18.4	12.1	7.9
[e]A IV-2	9.0	1.54	23.2	16.5	10.7
[e]A IV-3	9.0	2.00	29.6	22.9	11.5
NA IV-4		4.530	[h]258±9	[i]68±9	[i]15.0±2.9

[a]D, vacuum distillation (nickel crucible liner used except where noted); A, vacuum amalgamation; NA, neutron activation. Roman numeral identifies potassium series and the Arabic numeral identifies tube from which sample was taken.

[b]Environmental contamination study run.

[c]Cross-method analysis.

[d]Glass crucible liner used.

[e]Analyses performed by W. A. Dupraw and J. W. Graab using same apparatus.

[f]Appears in reference 15 as sample NASA-Cu-7.

[g]Environmental contamination: distillation, 270 mg of O/torr; amalgamation, 400 mg of O/torr. Method blank: distillation, 2.8 μg of O; amalgamation, 3.1 μg of O.

[h]Average value and standard deviation for multiple determinations on single sample: 3 determinations on series III potassium and 4 determinations on series IV potassium.

[i]Corrected only for oxygen found in container. Average value and standard deviation for multiple determinations on single empty container: 3 determinations for series III container and 4 determinations for series IV container.
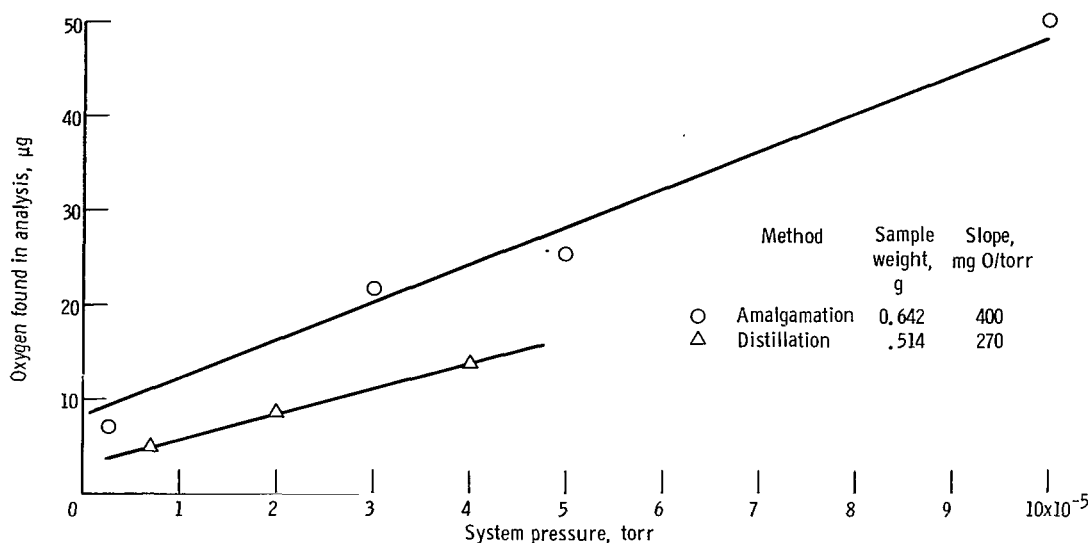
Figure 2. - Effect of air contamination on analytical results. Series I potassium.

A positive slope to the regression line would indicate a direct proportionality between contamination pickup and sample size, while a zero slope would indicate that environmental contamination pickup is independent of sample size over the range of experimental conditions covered. A t-test at the 5-percent significance level indicated that the slope of the regression line for both the distillation and amalgamation data did not differ significantly from zero.

Presumably, then, the environmental contamination pickup is a function only of the physical system (i.e., size and in-leak rate) and of the procedure (i.e., time, temperature, and exposed surface). Accordingly, the environmental correction factors of 270 and 400 milligrams of oxygen per torr for the distillation and amalgamation methods, respectively, were applied to the oxygen values found for the series II, III, and IV potassium.

The method blank (systematic errors other than the environmental contamination error) for the distillation and amalgamation methods was estimated for the series I potassium by plotting the oxygen values corrected for environmental contamination against sample size (fig. 3). The intercept of the least-squares line at zero sample weight yielded blank values of 2.8 and 3.1 micrograms of oxygen, respectively, for the distillation and amalgamation methods.

Corrections for the blank value found for the series I potassium were applied to the environmental-contamination-corrected values of the series II, III, and IV potassium. The oxygen values corrected for both systematic errors are listed in the fifth column of table I (p. 7). The oxygen concentrations were calculated as potassium monoxide (K_2O). A summary of results for the mean values of the oxygen found $\overline{X}$ and the standard deviation of the mean s for series I to IV potassium are given in table II.
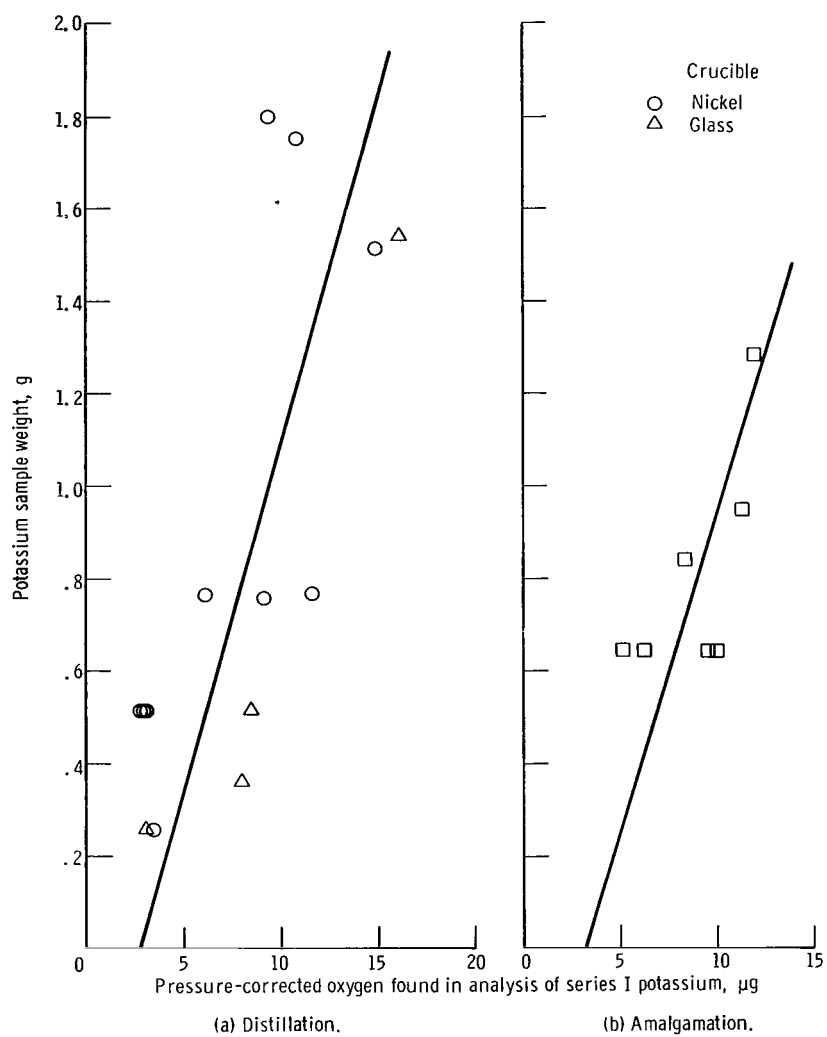
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(a) Distillation.   (b) Amalgamation.

Figure 3. - Method blank determination.

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TABLE II. – SUMMARY OF RESULTS FOR DETERMINATION

OF OXYGEN IN POTASSIUM

Method	Number of determinations	Mean oxygen found, $\overline{X}$, ppm	Standard deviation of mean, s
Series I potassium			
Distillation	14	[a]6.1	[b]4.7
Amalgamation	7	[c]7.2	[d]2.7
Series II potassium			
Distillation	7	8.7	2.7
Amalgamation	3	6.9	2.5
Series III potassium			
Distillation	6	16.1	4.8
Amalgamation	4	15.6	4.1
Neutron activation	[e]3	23.5	8.4
Series IV potassium			
Amalgamation	4	9.5	1.9
Neutron activation	[e]4	15.0	2.9

[a]Slope of least-squares line (fig. 3(a)).

[b]Standard deviation of least-squares line (fig. 3(a)).

[c]Slope of least-squares line (fig. 3(b)).

[d]Standard deviation of least-squares line (fig. 3(b)).

[e]See footnotes h and i, table I.

For the series I potassium, $\overline{X}$ and s were taken as equal to the slope and the standard deviation, respectively, of the least squares lines in figure 3 (p. 9).

DISCUSSION

Vacuum Distillation as Compared to Vacuum Amalgamation

The agreement between the mean values for the vacuum distillation and the vacuum amalgamation methods is very good. A comparison of the precision of results between the amalgamation and distillation methods was made both by comparing the variances (square of the standard deviations) of the individual series of potassium and by pooling variances of all series (refs. 19 and 20). At a significance level of 10 percent there was no difference in variability between methods. The pooled standard deviations for the vacuum distillation and vacuum amalgamation methods are 4. 3 and 3. 1, respectively.

As further confirmation of the agreement of mean values and precision between the amalgamation and distillation methods, the results obtained from cross-method analyses are pertinent. Three sample tubes, I-2, I-3, and II-1, were cross-method analyzed: part of the potassium in each tube was analyzed by the distillation method and part by the amalgamation method (see table I, p. 7). The mean oxygen values and standard deviations for the I-2, I-3, and II-1 tubes, 7. 6±3. 6, 7. 9±1.1, and 6. 3±1. 5 parts per million, respectively, are in good agreement with the values obtained (1) among tubes for either method and (2) between methods.

It is of interest to note that the vacuum amalgamation analyses were performed by two different teams of analysts using the same apparatus, series I potassium by one team and series II, III, and IV potassium by the other team. No significant difference between teams was observed in the precision of results or in the agreement with respective vacuum distillation mean values.

Neutron Activation as Compared to Vacuum Amalgamation

Agreement between the mean values for the neutron activation method and the vacuum amalgamation (or distillation) method is questionable. As a means of evaluating the significance of the observed difference in mean values between methods, three hypotheses can be proposed:

(1) There is no real difference between mean values.

(2) There is a real difference between mean values because of the presence of alkaline impurities other than potassium monoxide.

(3) There is a real difference between mean values because of undetected systematic errors.

Hypothesis 1 can be tested statistically (ref. 19). A comparison of the mean analytical values of the series IV amalgamation $\overline{X}_A$ and neutron activation $\overline{X}_N$ indicates that at a 10-percent significance level hypothesis 1 is not correct. A comparison of the mean values for the series III amalgamation and neutron activation indicates that at a 10-percent significance level hypothesis 1 is correct. However, any final decision concerning the hypothesis must be qualified by the β error, the probability of failing to find a difference that really exists. For the series III comparison the β error is 0.5. All in all, hypothesis 1 should probably be rejected.

The plausibility of hypothesis 2 can be tested by a comparison of the oxygen values found with an estimate of the values predicted when alkaline impurities other than potassium monoxide are present in the potassium. Potential impurities of consequence in potassium are potassium monoxide (K_2O), potassium peroxide (K_2O_2), potassium superoxide (KO_2), potassium hydroxide (KOH), potassium carbonate (K_2CO_3), potassium hydride (KH), and potassium carbide (K_2C_2).

It should be recognized that none of the methods investigated herein are specific for K_2O. The amalgamation and distillation methods culminate in the isolation of mercury insoluble and nonvolatile alkaline residues, respectively, followed by alkalimetry of the residue. It is then tacitly assumed that K_2O is the sole alkaline impurity measured in these residues. Because the neutron activation method measures oxygen atoms (and not oxygen compounds) within a sample, the presence of potassium-oxygen species other than K_2O should result in neutron activation oxygen values that are larger than amalgamation or distillation method values. On the other hand, the presence of KH or K_2C_2 should yield the opposite result.

The magnitude of the discrepancy in analytical results between methods can be readily calculated as a function of both the species and the amount of alkaline impurity assumed to be present in a sample. Designating the amalgamation and neutron activation method "oxygen" values by V and V(1 + f), respectively, simultaneous equations were set up and solved to yield the mole ratio R, the ratio of K_2O to other alkaline impurities, as a function of the discrepancy factor f. In figure 4 are plotted curves $[K_2O]/[Y]$ against f, where Y is KOH, K_2CO_3, KH, and K_2C_2. Curves for the case where Y represents impurities such as KO_2 or mixtures of impurities, for example, KOH and K_2CO_3, would fall between the two limiting curves where Y is K_2CO_3 and K_2C_2.

For purposes of comparison, the observed difference in the mean values $\overline{X}_N - \overline{X}_A$ and the 90-percent confidence interval for the true difference between mean values $m_N - m_A$, in terms of f, are shown in figure 4 for the series III and IV neutron activation and amalgamation analyses. It is apparent that the presence of alkaline impurities other than K_2O in the potassium could reasonably account for the difference in oxygen
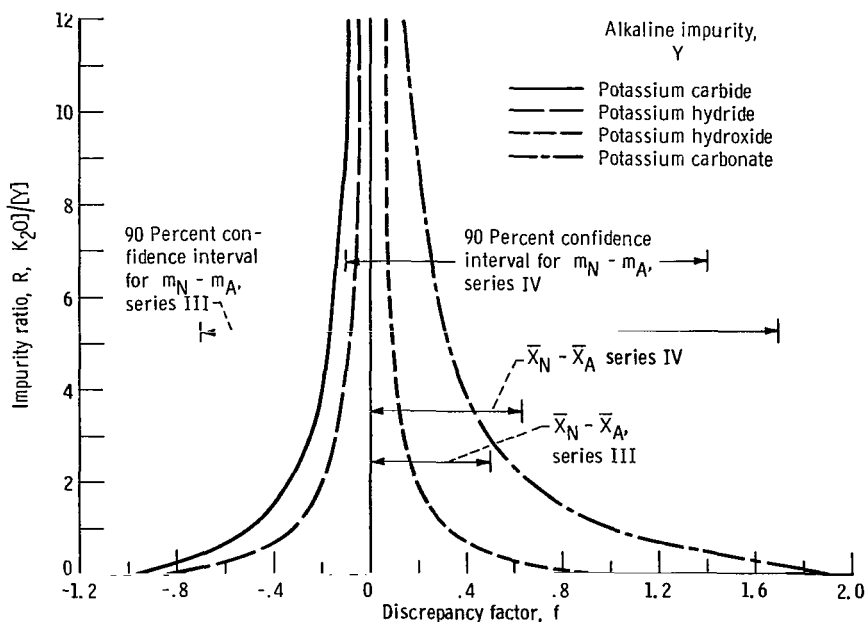
Figure 4. - Plot of impurity ratio against discrepancy factor.

values between methods.

Further support of the validity of hypothesis 2 was found in supplemental tests, which evidenced the presence of K_2CO_3 in the potassium samples. The residues from six vacuum distillation runs on series I and III potassium were double titrated with standard acid to the phenolphthalein and the methyl red end points as rapidly as possible to minimize atmospheric carbon dioxide (CO_2) contamination. From the results of these titrations it can be estimated that the approximate value of R, where Y is K_2CO_3, is 6. Because atmospheric CO_2 was certainly absorbed during the titration, this value of R must be considered as a limiting value, that is, $R \geq 6$. From the appropriate curve in figure 4, this limiting value of R predicts a discrepancy factor of 0.26.

The validity of hypothesis 3 can be reasoned from the observed effect of environmental contamination reported herein (fig. 2, p. 8). The preparation of samples for neutron activation analyses involves first the transfer of potassium from sample tubes to capsules and last the sealing of the capsule. These operations for series III and IV were performed either in vacuum ($\sim 1 \times 10^{-5}$ torr) or in a low-impurity inert-gas environment, respectively. In either instance, some contamination of the potassium is predictable.

For the capsule filled and sealed in vacuum, a correction can be estimated from the amalgamation method contamination correction value of 400 milligrams of oxygen per torr. This may be considered as a limiting contamination correction, since the time required to fill and seal the neutron activation capsule is less than the exposure time

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experienced during the amalgamation analyses. When applied to the series III neutron activation value 23.5 parts per million, the limiting contamination correction yields a mean oxygen value of $\bar{X}_{N_{corr}}$ of 19.7 parts per million and a corresponding f of 0.26 for $\bar{X}_{N_{corr}} - \bar{X}_A$.

On the basis of the foregoing discussion of hypotheses 1 to 3, it can be argued that the differences between the neutron activation and the amalgamation mean oxygen values are real, and due in part to the presence of alkaline impurity other than K_2O and in part to unaccounted-for atmospheric contamination. Consequently, the application of the aforementioned estimated limiting value corrections should enhance the agreement of results between methods.

For the series III and IV potassium, when the estimated adjustment for alkaline impurity (as K_2CO_3) is applied to the oxygen values, the difference between neutron activation and amalgamation method mean oxygen values is reduced by a factor of about 2. When, in addition, an estimated correction for environmental contamination is applied to the series III potassium, the difference in fully adjusted mean oxygen values becomes vanishingly small. (A comparable contamination correction could not be applied to the alkaline impurity-adjusted oxygen values of the series IV potassium for lack of data on atmospheric contamination pickup in the "inert" gas environment.)

CONCLUSION

When corrected for systematic errors and the presence of alkaline impurities other than K_2O, the values for oxygen in potassium at concentrations less than 20 ppm obtained by the vacuum distillation, vacuum amalgamation, and neutron activation methods show good agreement. Since agreement of results between analytical methods may be used as an index of accuracy, it can be concluded that the agreement of results from three independent analytical methods establishes the accuracy of the oxygen determinations reported herein with a high degree of confidence.

Lewis Research Center,
    National Aeronautics and Space Administration,
        Cleveland, Ohio, November 10, 1965.

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